MATERIALS CHARACTERIZATION SERIES

SERIES EDITORS: C. Richard Brundle and Charles A. Evans, Jr.

材料表征原版系列丛书

光学材料的表征

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Gregory J. Exarhos

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EDITOR

Gregory J. Exarhos

SERIES EDITORS

C. Richard Brundle and Charles A. Evans, Jr.

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MATERIALS CHARACTERIZATION SERIES
Surfaces, Interfaces, Thin Films

Series Editors: C. Richard Brundle and Charles A. Evans, Jr.

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Characterization of Optical Materials, Gregory J. Exarhos

Characterization of Tribological Materials, William A. Glaeser

Characterization of Organic Thin Films, Abraham Ulman

Preface to the Reissue of the Materials Characterization Series

The 11 volumes in the *Materials Characterization Series* were originally published between 1993 and 1996. They were intended to be complemented by the *Encyclopedia of Materials Characterization*, which provided a description of the analytical techniques most widely referred to in the individual volumes of the series. The individual materials characterization volumes are no longer in print, so we are reissuing them under this new imprint.

The idea of approaching materials characterization from the material user's perspective rather than the analytical expert's perspective still has great value, and though there have been advances in the materials discussed in each volume, the basic issues involved in their characterization have remained largely the same. The intent with this reissue is, first, to make the original information available once more, and then to gradually update each volume, releasing the changes as they occur by on-line subscription.

C. R. Brundle and C. A. Evans, October 2009

Preface to Series

This Materials Characterization Series attempts to address the needs of the practical materials user, with an emphasis on the newer areas of surface, interface, and thin film microcharacterization. The Series is composed of the leading volume, *Encyclopedia of Materials Characterization*, and a set of about 10 subsequent volumes concentrating on characterization of individual materials classes.

In the *Encyclopedia*, 50 brief articles (each 10 to 18 pages in length) are presented in a standard format designed for ease of reader access, with straightforward technique descriptions and examples of their practical use. In addition to the articles, there are one-page summaries for every technique, introductory summaries to groupings of related techniques, a complete glossary of acronyms, and a tabular comparison of the major features of all 50 techniques.

The 10 volumes in the Series on characterization of particular materials classes include volumes on silicon processing, metals and alloys, catalytic materials, integrated circuit packaging, etc. Characterization is approached from the materials user's point of view. Thus, in general, the format is based on properties, processing steps, materials classification, etc., rather than on a technique. The emphasis of all volumes is on surfaces, interfaces, and thin films, but the emphasis varies depending on the relative importance of these areas for the materials class concerned. Appendixes in each volume reproduce the relevant one-page summaries from the *Encyclopedia* and provide longer summaries for any techniques referred to that are not covered in the *Encyclopedia*.

The concept for the Series came from discussion with Marjan Bace of Manning Publications Company. A gap exists between the way materials characterization is often presented and the needs of a large segment of the audience—the materials user, process engineer, manager, or student. In our experience, when, at the end of talks or courses on analytical techniques, a question is asked on how a particular material (or processing) characterization problem can be addressed the answer often is that the speaker is "an expert on the technique, not the materials aspects, and does not have experience with that particular situation." This Series is an attempt to bridge this gap by approaching characterization problems from the side of the materials user rather than from that of the analytical techniques expert.

We would like to thank Marjan Bace for putting forward the original concept, Shaun Wilson of Charles Evans and Associates and Yale Strausser of Surface Science Laboratories for help in further defining the Series, and the Editors of all the individual volumes for their efforts to produce practical, materials user based volumes.

C. R. Brundle and C. A. Evans, Jr.

Preface to the Reissue of *Characterization of Optical Materials*

Though there have been advances in the materials and in the design and manufacturing of integrated optical devices since this volume was originally published, the basic principles of the techniques and protocols for analysis and characterization of optical materials discussed here remain valid. There have, of course, been technical advances for many of these techniques (ease of use, software, spatial resolution, and extension of wavelengths into the UV for optical methods). Applications in the new, carbon-based materials, such as nanotubes and graphene, have developed. After this re-issue in a form close to the original, it is our intent that updates, covering the advances that have occurred, will be released as downloads as they become available.

C. R. Brundle and C. A. Evans, December 2009

Preface

The design and manufacture of advanced optical materials have been driven by a multidisciplinary approach from which new components and integrated optical devices have evolved. The diversity of optical materials—including metals, polymers, glasses, ceramics, semiconductors, and composites—provides a challenge to the analyst charged with characterizing optical surfaces and interfaces and developing associated structure–property relationships. Analysis of most optical materials must include not only measurements of optical properties, but a determination of the fundamental surface and interfacial material properties as well. For example, correlating the optical response of a material with its microstructure, residual interfacial stress, phase purity, and surface-roughness can lead to a refinement of processing methods in order to secure the optimum material for a particular application.

This volume—one component of the *Materials Characterization Series: Surfaces, Interfaces, Thin Films*—focuses on the kind of information derived from the principal analytical methods currently used to characterize optical materials. This information is useful for identifying the key parameters that control the optical response of a material. The theory and methodology of the analytical methods used for the surface and interfacial characterization of optical materials are discussed in the lead volume of the series, *Encyclopedia of Materials Characterization*, and are summarized in the appendix of this book. The intent of this volume is not to discuss instrumental methods in great detail, but to provide the necessary background information to permit a constructive dialogue to be initiated between the researcher with a specific problem to address and the technical specialist skilled in applying a particular method.

This book consists of two major sections, an introductory chapter, and an extensive appendix, which summarizes the analytical methods pertinent to the characterization of optical materials. The book covers both crystalline and amorphous materials—with applications from the far infrared (~1 mm wavelength) to the vacuum ultraviolet (~100 nm wavelength) regions of the spectrum—and presents a succinct discussion of the type of information obtainable using various key surface characterization methods. The extensive list of references for each chapter may be consulted to gain a more thorough understanding of a particular subject area.

The topics covered and organization of this volume grew from a number of discussions with Marjan Bace of Manning Publications Company, who persuaded me to consider editing a text on the surface and interfacial characterization of optical materials. His direction and advice were greatly appreciated. I wish to express my gratitude to all contributing authors, who took time from their busy schedules to participate in writing this volume. I hope the readers will find our

efforts of practical value. Our Managing Editor, Lee Fitzpatrick, also of Manning Publications Company, was instrumental in persuading the authors to submit their chapters in a timely manner so that the production schedule could be met. Her efforts in dealing with all involved were invaluable. Finally, I wish to acknowledge the Pacific Northwest Laboratory, operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RLO 1830, for allowing me the time and resources to complete this project.

Gregory J. Exarhos

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Introduction

GREGORY J. EXARHOS

The development of improved optical materials requires a thorough understanding of their surface and subsurface structure and associated chemistry so that optical properties can be modified in a controlled manner and the materials durability enhanced. Advanced surface analytical methods are used to probe these materials to discern structure–property relationships which are used for designing materials with a specific response. This volume is intended to provide the researcher an introduction to the principal methods used for the surface and interfacial analysis of materials used for optical applications.

Optical materials discussed include bulk solids, thin films, and multilayer dielectric coatings which find use in applications requiring reflection, refraction, absorption, emission, scattering, or the diffraction of infrared, visible, or ultraviolet light having wavelengths from about 100 nm to 10 mm. Included in the variety of materials covered by this definition are metals, glasses, polymers, semiconductors, ceramic oxides, carbides and nitrides, and diamond. A cadre of surface analytical techniques is required for the characterization of such a diverse collection of materials. Furthermore, methods used to analyze a specific class of optical materials may be entirely inappropriate for a different materials class. Once an appropriate analysis method or methods have been identified, the information derived from the measurements can be used in several ways, such as assuring that the optical material or device meets desired specifications and controlling interactively processing parameters during manufacture. In a more fundamental approach, empirical models that relate structural properties of the material and chemical bonding to the intrinsic optical response can be developed and refined. Such models provide a basis for optimizing processing parameters to achieve a targeted optical response. The goal of this volume is to review the most important methods for characterizing the surface and interfacial properties of optical materials and to demonstrate for each method the kind of information obtained and how to interpret it.

The surface and interfacial properties of an optical material can be probed at length scales ranging from micrometers to tenths of nanometers. Microscopic defects which control surface smoothness and coating homogeneity contribute to the scattering of light and associated degradation of the optical response. Likewise, structural perturbations at the atomic level can alter optical properties through modifications to the complex refractive index of the material. To characterize *defects* at this level, high-resolution diffraction measurements coupled with the evaluation of localized chemical bonding using molecular spectroscopic techniques may be required. Such an approach can provide information regarding the structural phase of the material, the degree of surface/interfacial stress present, and the nature of the chemical bonding associated with a particular structure. Therefore, analytical probes having sensitivity at increasing levels of spatial and depth resolution are required in order to understand the physical and chemical properties that control the associated optical response of the material. These issues are addressed in Part I of this volume where correlations between the optical response of a material, surface morphology, and associated microstructure are described.

Surface roughness is a critical parameter that can degrade the performance of optical materials through light scattering processes. In addition, modification to the optical response can result from rough surfaces in which chemical attack from the ambient environment (water) is accelerated due to the increased surface area. The assessment of surface roughness is therefore critical to the behavior and long term stability of optical materials. Surface topology is a recurrent theme throughout this volume beginning with its introduction and definition in the first chapter and continuing in later chapters, including those that focus on diamond coatings and laser damage phenomena.

A powerful technique for characterizing the optical response of materials relies on measuring the change in the properties of light reflected from a surface or interface. Ellipsometry has evolved into a powerful noninvasive surface characterization tool, and its application to studies of optical materials is reviewed in this volume. Instruments are currently available that directly interface to vacuum chambers for real-time in situ characterization during the deposition of optical coatings or modification of surfaces through ion irradiation. Data acquired using this technique can be interpreted on the basis of various empirical models (Effective Medium Approximation) designed to extract microstructural information intrinsic to the surface under investigation. Such measurements complement the electron beam microscopy techniques used to evaluate microstructure but have the advantages of being nondestructive and amenable to materials analysis in real time. Ellipsometric measurements are relatively easy to perform, but considerable effort is required to interpret the measured data. Chapter 2 provides insight into the kinds of information that can be extracted from ellipsometric measurements of optical surfaces and indicates limitations of the technique. The quality of information inferred from these optical reflection methods is strongly dependent upon the model used to interpret the data. New algorithms have been developed relating the microstructure of a material to the optical response. One example, based upon a finite element model of the microstructure in a thin film, appears in a recent article by Risser and Ferris.[1] The technique of using ellipsometric methods to provide more extensive microstructural information about surfaces and interfaces will continue to mature as the models used to interpret the data become more refined.